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No. III.

Analyses of the Chrysoberyls from Haddam and Brazil. By Henry Seybert.—Read, 5th March, 1824.

IN the summer of 1823, I visited Haddam, in the State of Connecticut. Among the various substances there collected, was the Chrysoberyl, a mineral much esteemed on account of its rarity. It occurs disseminated in a coarse grained granite, in which the predominant ingredient is a white feldspar, which Professor Berzelius regards as *Albite*, perfectly resembling that of Finbo. In the same granite this celebrated chemist observed the *Columbite*.* It is also associated with greyish quartz, manganesian garnet of a fine blood red colour, and a yellow granular substance, which some mineralogists supposed to be a variety of the cymophane; but from its inferior hardness and general chemical composition, I recognised it to be common beryl.

For the earliest chemical information concerning the Chrysoberyl, we are indebted to Professor Klaproth. He published his analysis of it in 1795,† and gave the following constituents of it, viz. alumina, 71.50; lime, 6.; oxide of iron, 1.50; silica, 18.; loss, 3. Berzelius presented us with a formula founded on this composition;‡ but from his experiments with the blowpipe he was led to conclude that it contained no lime, and that it was a subsilicate of alumina.§ In this he was apparently confirmed by Professor Thom-

* Essai de l' Emploi du Chalumeau, p. 243.

† Beitrage, vol. i. p. 97.

‡ Systeme de Mineralogie, p. 219,— $C^4S + 18A^4S$.

§ Essai de l' Emploi du Chalumeau, p. 325.

son,* who quotes Klaproth's analysis, and states that he examined the mineral some years ago, but having accidentally lost his results, he was unable to publish them. He observes, however, that the only constituents he found were alumina, silica, and oxide of iron. When I was about to prepare the communication which I now have the honour to lay before the Society, a more recent analysis of the Chrysoberyl of Brazil, by M. Augustus Arfwedson, was observed, by me, in Tilloch's Philosophical Magazine.† He confirmed the results of Professor Thomson, and considered the chemical composition of this substance to be—silica, 18.73; and alumina, 81.43, with a trace of oxide of iron.

The cymophane, from Haddam, was sent to M. Haüy by the late Dr. Bruce, in 1810, to have his opinion concerning its nature.‡ Previous to that period, the mineralogists in the United States supposed it to be *Corundum*. The late celebrated crystallographer observes, "La cymophane des Etats Unis a d'abord été prise pour une variété de corindon. Effectivement elle se rapproche de ce mineral par sa dureté, par sa pesanteur spécifique, et même par le resultat de son Analyse, qui a donné environ 72 parties d'alumine sur 100, avec 18 de silice, et 6 de chaux."§ I was anxious to examine the cymophane found at Haddam, especially as M. Haüy does not name the author of the analysis he quotes. The specimen used for my experiments was of a pale green colour. It did not present any of the chatoyant appearance so remarkable in the variety from Brazil, and some specimens from Saratoga in New York, where it was lately discovered by Dr. Steel. Its specific gravity, by two trials, was 3.508 and 3.597. It is not magnetic, and before the blow-pipe it is infusible. For a further description of the physical characters of this mineral, I refer to Haüy and Cleaveland.

* Thomson's Chemistry, vol. iii. p. 213.

† No. for November, 1823, p. 357.

‡ Annales du Museum d'Histoire Naturelle, tome xviii. p. 57.

§ Traité de Mineralogie, 2me Edition, vol. ii. p. 309.

Three grammes of the mineral were examined under the impression that Professor Klaproth's analysis was accurately made. It was decomposed in the usual manner with four parts of caustic potash, and subsequently treated with diluted muriatic acid ; but the solution was imperfect. The insoluble matter was collected on a filter, and it amounted to 25 or 30 per 100. It was repeatedly acted on in the same way, and each time it diminished in quantity, until the fourth experiment. It then weighed about fifteen-hundreths, and thereafter resisted all further efforts to render it soluble by these means. This residue was then boiled in concentrated sulphuric and muriatic acids, but neither of them dissolved more than one-third of it. These solutions were tested by different re-agents, and greatly to my surprise, the addition of subcarbonate of ammonia occasioned a flocculent precipitate, which entirely re-dissolved in an excess of the alkaline subcarbonate. I immediately suspected the presence of *Glucina*, but was much at a loss to explain its insolubility, until I observed Berzelius's analysis of the *Euclase*,* in which he met with a compound of glucina and oxide of tin that obstinately resisted acids. He also met with refractory combinations of this earth and the oxides of manganese and cerium. I next endeavoured to dissolve the compound by the acid sulphate of potash ; but this method did not succeed. I was not more successful with the nitric and nitromuriatic acids ; nor could it be dissolved by means of boric acid. Berzelius having discovered columbium in the gangue of the cymophane from Haddam, the insoluble residue was tested for the oxide of that metal, but all my attempts were fruitless. At length, I supposed, that as barytes could be brought into contact with this substance more conveniently than potash at a high temperature, it might decompose it. With this view, a portion of the insoluble matter was exposed to a strong heat, during one hour with six parts of nitrate of barytes in a platina crucible.

* Nouveau Systeme de Mineralogie, p. 289.

The calcined mass was boiled in nitric acid. In this way nearly two-thirds of the matter that could not be entirely attacked in any other way, were dissolved. The same treatment was repeated, until nearly the whole of it was taken up, which happened after the fourth calcination. It was then no further acted on.

After making numerous experiments on the matter that resisted nitrate of barytes and nitric acid, I ascertained, that it was not acted on by alkalies nor acids when used separately, but after having been previously calcined with caustic potash, it readily dissolved in muriatic acid, yielding a solution of a pale yellow colour, which gave a reddish precipitate with an infusion of galls, a deep green precipitate with the hydrosulphate of potash, and a white precipitate with alkalies. Hence it was oxide of titanium.

After the barytes was separated with sulphuric acid, the nitric solutions were united, and treated with an excess of subcarbonate of ammonia. An abundant precipitate ensued, which entirely re-dissolved in the excess of subcarbonate. By ebullition it was again precipitated, and when calcined, it was in the form of a light white powder, possessing all the properties that characterise *Glucina*. With the sulphuric and muriatic acids it formed very sweet astringent deliquescent salts. By caustic potash it was precipitated from its solutions, and the precipitate re-dissolved in the excess of the alkali. Klaproth and Arfwedson, in their analyses of the Chrysoberyl from Brazil, considered the insoluble matter remaining after they had treated the mineral with potash and muriatic acid, to be *Silica*. This will explain why their results differ so essentially from mine.

After having thus satisfied myself of the composition of the residue above mentioned, I resumed my preliminary experiments, and proceeded to examine the muriatic solution obtained from the treatment of the mineral with potash and muriatic acid. From this solution some silica was separated. A portion of the liquid was treated with caustic ammonia, and then tested for *Lime* with oxalate of potash, but none

of it could be detected. To the remaining liquor a considerable excess of subcarbonate of ammonia was added, and the precipitated matter was digested twenty-four hours. It was then separated by filtration, and the fluid was boiled till all the ammonia was expelled. No glucina was thus precipitated. Hence we conclude, that the very small portion of titanium above mentioned, rendered the whole of the glucina so refractory. The alumina precipitated by the subcarbonate of ammonia was mixed with a small quantity of oxide of iron. It was soluble in caustic potash, and with this alkali and sulphuric acid it gave regular octædral crystals of alum. The liquor, when tested with phosphate of soda and ammonia, was found to contain *no Magnesia*.

After the preliminary experiments, I commenced the following

ANALYSIS OF THE CHRYSOBERYL FROM HADDAM.

A. Five grammes of the mineral, reduced to small fragments in an iron mortar, were carefully porphyrised in one of agate, from which it acquired the additional weight of 0.13 grammes. The 5.13 grammes were then exposed to a red heat, and thereby suffered a diminution of 0.40 per 100.

B. The calcined mineral (*A*) was heated, during one hour, in the silver crucible, with caustic potash, and the product was treated with diluted muriatic acid; the solution was of a lemon yellow colour. There remained a white insoluble residue, which after calcination weighed 1.47 grammes. It was repeatedly calcined with caustic potash, and treated with diluted muriatic acid, with the following results:

After the 2d experiment, it weighed 0.97 grammes.

3d	0.89
4th	0.85

By the fifth treatment it was not diminished, and then presented itself in the form of a light white powder, resembling pure silica in appearance.

C. The residue (*B*) was repeatedly strongly calcined with six parts of nitrate of barytes, and subsequently boiled with nitric acid.

After the 1st treatment, there remained 0.43 grammes.

2d 0.15

3d 0.06

And by the 4th operation only 0.01 gramme was dissolved.

The remaining 0.05 grammes were essayed in the manner related in the preliminary experiments, and thus proved to be oxide of titanium. Hence we have 1. per 100 of that oxide.

D. The nitric solutions were united and evaporated to dryness, to expel the excess of the acid. The saline mass was dissolved in water, and after the barytes was separated with sulphuric acid, an excess of subcarbonate of ammonia was added to the solution. An abundant precipitate appeared, which entirely re-dissolved. The *Glucina* was precipitated by ebullition. Afteredulcoration and calcination, it weighed 0.79 grammes, or 15.80 per 100.

E. The several muriatic solutions (*B*) were united and evaporated to a dry mass, which was treated with muriatic acid, and there remained 0.33 grammes of silica, from which deduct 0.13 grammes acquired from the agate mortar; and there will be 0.20 grammes, or 4. per 100 as a constituent of the mineral.

F. After the silica was separated from the liquid (*E*), the alumina and oxide of iron were precipitated by means of a great excess of subcarbonate of ammonia. After twenty-four hours, the liquor was separated from the yellowish pre-

cipitate, and was boiled, but no *Glucina* was precipitated from it. The matter precipitated by the subcarbonate of ammonia consisted of 3.68 grammes of alumina, or 73.60 per 100., and 0.19 grammes of peroxide of iron, which, on account of the colour of the mineral, must be estimated as protoxide. The 0.19 grammes of peroxide are equivalent to 0.169 of protoxide, or 3.38 per 100.

THE CONSTITUENTS OF THIS CHRYSOBERYL THEREFORE
ARE,

(Per 100 parts)

<i>A.</i>	Moisture	0.40
<i>C.</i>	Oxide of titanium	1.00
<i>D.</i>	Glucina	15.80
<i>E.</i>	Silica	4.00
<i>F.</i>	Alumina	73.60
<i>F.</i>	Protoxide of iron	3.38
		<hr/>
		98.18
		<hr/>
		100.00
		<hr/>
Loss		1.82

As the preceding results differed so essentially from the analyses of the chrysoberyl from Brazil by Klaproth and Arfwedson, I determined to examine a specimen from that locality. 1.5 grammes were analysed in the manner above mentioned, and the following results were obtained :—

(Per 100 parts)

Water	0.666
Oxide of titanium	2.666
Glucina	16.000
Silica	5.999
Alumina	68.666
Protoxide of iron	4.733
	<hr/>
	98.730
	100.000
	<hr/>
Loss	1.270

In estimating these constituents according to the electro-chemical theory, I believe that the oxide of titanium, notwithstanding its important agency in the analytical experiments, must be regarded as an accidental ingredient, as well as the oxide of iron, which in some measure may have been derived from the iron mortar. As the cymophane of Brazil appears to be constituted more conformably to the hypothesis of chemical proportions than that of Haddam, the following calculation may be made, founded on its composition, which gives for the essential constituents of Chrysoberyl,

(Per 100 parts)

Silica	6.61	containing Oxygen	3.32
Alumina	75.75		35.38
Glucina	17.64		5.49

and very nearly corresponds with the following mineralogical formula, A^4S+2GA^4 .